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14. ABSTRACT The development of an understanding of the tribological properties of carbon based coatings/materials will ultimately provide unique materials needed in the successful implementation of future MEMs devices. MEMs devices, in particular radio frequency MEMs, are viewed as being a promising technology for implementation of future advanced communication devices.					
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**Final Report**

**AFOSR Project:**

**Uncovering the fundamental nature of tribological interfaces:  
High-resolution tribology and spectroscopy of ultrahard nanostructured diamond films for  
MEMS and beyond**

**University of Pennsylvania  
Philadelphia, PA 19104**

**Reporting Date: Mar. 15, 2005 – Dec. 31, 2007**

**Project#: FA9550-05-1-0204**

**Principal Investigator: Prof. Robert W. Carpick  
Mechanical Engineering and Applied Mechanics Dept., University of Pennsylvania  
(previously at the University of Wisconsin-Madison)**

**Co-Investigators: Prof. P.U.P.A. Gilbert (formerly De Stasio)  
Physics Dept., University of Wisconsin-Madison**

**Dr. Anirudha V. Sumant  
Argonne National Laboratories  
(previously at the University of Wisconsin-Madison)**

20130918437

## Statement of Objectives

The original objectives of the proposal were as follows:

1. Obtain high-quality ultrananocrystalline diamond (UNCD) and tetrahedral amorphous carbon (ta-C) films from the collaborators. The films will be deposited on substrates and on AFM tips and nitride balls for use in a fretting wear device.
  - a. Undoped UNCD and UNCD doped with different amounts of nitrogen will be studied
  - b. ta-C samples subjected to different stress-relieving annealing procedures will be studied
2. Treat samples with different surface modification treatments, specifically hydrogen and fluorine termination.
3. Carry out AFM and fretting wear measurements of frictional sliding for self-mated interfaces of these films. Evaluate the regimes of wearless sliding, the onset of wear, and the progression of wear.
4. Evaluate wear of the AFM tips by high resolution TEM/EELS microscopy/spectroscopy.
5. Evaluate wear of the substrates and coated balls by high resolution PEEM/NEXAFS microscopy/spectroscopy.

The objectives were subsequently revised as follows:

1. In addition to investigating UNCD and ta-C, also investigated are (a) diamond-like carbon (DLC) grown at UW-Madison, and at Argonne National Laboratory, and (b) nanocrystalline diamond grown at UW-Madison, and by Advanced Diamond Technologies Inc.
2. Because of this expanded emphasis on materials, investigation of the nitrogen-doped UNCD films and of the effect of fluorine termination were held off.
3. Because of exciting and interesting results that were obtained, the AFM Investigations were restricted to the wearless regime and the onset of wear for the AFM measurements.
4. Not modified.
5. Preliminary PEEM and AFM investigations of wear behavior in polysilicon MEMS devices from Sandia National Laboratories were also carried out. These devices are coated with a fluorinated monolayer for to reduce adhesion and wear. This is a widely-used coating in the MEMS industry and represents a possible solution to tribological problems in existing silicon-based MEMS.



## Abstract

Several new and significant fundamental insights into the tribological behavior of ultrastrong carbon-based films with extremely low friction and wear have been uncovered. Specific limits on this low friction and wear behavior were elucidated. In addition, photoemission electron microscopy (PEEM) has been successfully established as a powerful tool for understanding the changes that occur due to tribological sliding. By using PEEM, atomic force microscopy, and a range of surface science techniques, the tribology of carbon-based materials has been brought to a new level of understanding. The new insights include the paradigm-shifting demonstration that the low friction of diamond films is not due to the formation of graphite at the sliding interface as widely thought, but rather due to the passivation of dangling bonds by dissociative adsorption of species such as water. More generally, adhesion (at the nano-scale), friction (both at the nano- and macro-scale) and wear (at the macro-scale) in carbon-based films has been shown to be strongly affected by the composition of the topmost layer of atoms at the surfaces, as controlled by the environment and the sliding conditions.

Two graduate students were recruited to the project and fully trained on the PEEM and AFM instrumentation. One has defended his Ph.D. thesis (2007). A staff scientist was also partially funded on this effort. Undergraduate students were also supported. Together, they performed multiple PEEM experiments on wear tracks on carbon-based films and polysilicon micro-electro mechanical systems (MEMS) devices, a comprehensive set of AFM measurements of the nanotribology of carbon-based materials, and TEM measurements of wear at the nanoscale.

These studies included small-amplitude wear tracks on ultrananocrystalline diamond (UNCD), tetrahedral amorphous carbon (ta-C), and diamond like carbon (DLC), wear in a polysilicon MEMS device known as the "nanotractor", and studies of the structure and composition of UNCD, ta-C, and nanocrystalline diamond (NCD) films. They also carried out extensive AFM investigations of the nanotribological properties of UNCD, ta-C, and DLC. They have discovered that UNCD wear leads to both oxidation and a small amount of rehybridization on the surface. This passivation of the surface by oxidation is largely responsible for the low friction. In ta-C, surprisingly, an increase in the diamond ( $sp^3$ -bonded carbon) content of the worn area is observed. This is attributed to the removal of a weakly-bound  $sp^2$ -rich layer by the sliding action. In the nanotractor, wear is characterized by a dramatic level of oxidation of silicon, and partial removal and damage of the monolayer coating. These insights point the way toward further experiments to quantify the wear mechanisms at play.

Results obtained are now published in a total of 9 peer-reviewed publications (including one in *Science* and one in *Phys. Rev. Lett.*) and 2 invited, peer-reviewed review articles, with 4 more articles in preparation. Results from this study were the subject of 27 invited presentations and 14 contributed presentations at national and international meetings, universities, workshops, and industry venues. Several of the publications garnered press in the national science media.

## Accomplishments/New Findings

New fundamental insights were gained into how differences in bonding structure, film micro/nanostructure, surface chemistry, and chemical environment produce different nanotribological behavior in carbon-based films. Several distinct carbon-based thin film materials were investigated: ultrananocrystalline diamond (UNCD), nanocrystalline diamond

(NCD), tetrahedral amorphous carbon (ta-C), and diamond-like carbon (DLC). To understand the surface chemistry of these materials, near-edge x-ray absorption fine structure (NEXAFS) spectroscopy was used to determine the chemical composition and the nature of the bonds in the near-surface region. To study the corresponding nanotribological properties, the atomic force microscope (AFM) was used to measure the work of adhesion and frictional forces between diamond AFM tips and the materials of interest.

Friction and wear measurements were performed using a reciprocating tribometer apparatus for UNCD and ta-C films in contact with both UNCD-coated and  $\text{Si}_3\text{N}_4$  spheres, in ambient and controlled-atmosphere conditions. The micron-scale wear tracks produced on these films were analyzed by atomic force microscopy (AFM), optical profilometry, Raman microscopy, and photoelectron emission microscopy (PEEM) to obtain quantitative information on structural modifications and chemical changes inside the wear track. The ability of the PEEM spectromicroscopy technique to spatially resolve ( $\sim 10$  nm, ideally) and chemically characterize regions of interest is unparalleled and therefore ideally suited for this work. The results show for the first time that it is possible to detect chemical changes with great detail occurring within the micro-scale wear track of these materials. Furthermore, correlations were established between spectroscopically-identified chemical changes within the wear track and corresponding changes in the friction coefficient recorded during tests. In the case of a UNCD film in contact with a  $\text{Si}_3\text{N}_4$  ball,  $\text{SiO}_x$  complexes form within the wear track, and correspondingly the friction coefficient increases. In the case of a ta-C film in contact with  $\text{Si}_3\text{N}_4$  ball, a decrease in friction coefficient was observed after annealing, and complex changes in the carbon bonding configuration on the surface occurred.

It was found that the nanotribological properties depend sensitively on the surface chemistry and bonding. In the case of UNCD, the work of adhesion can be lowered between a diamond tip and the UNCD surface by terminating the surface with hydrogen. The work of adhesion of a UNCD surface is already very low ( $\sim 60$  mJ/m<sup>2</sup>). However, the adhesion can reach the van der Waals' limit ( $\sim 10$  mJ/m<sup>2</sup>) after hydrogen termination. Comparing that to the work of adhesion for a self-mated silicon interface ( $> 800$  mJ/m<sup>2</sup>), the adhesion and friction are significantly lower. These results are further proof that the structure and chemistry at the surfaces of an interface have an immense effect on the tribological behavior, and, in this case, with some careful thought they can be modified to reduce adhesion.

A separate thrust has involved the fabrication of monolithic UNCD and ta-C AFM cantilevers. Minimizing wear on AFM tips is a critical challenge. UNCD and ta-C have excellent mechanical and tribological properties that are ideally suited for AFM probes, such as low macro-scale wear and friction, high hardness, and chemical inertness. This work has accomplished the first successful batch fabrication of monolithic probes from both materials. An array of pyramidally-etched pit molds was formed on a silicon wafer by anisotropic etching in KOH. Oxidation sharpening of the molds was performed, followed by deposition of UNCD or ta-C films  $\sim 1$ -3  $\mu\text{m}$  thick. The coated wafers were then subjected to metal mask deposition, photolithography, and oxygen reactive ion etching to form monolithic cantilevers with integrated pyramidal tips. Individual probes were then released and physically bonded to a holding substrate for testing. SEM, TEM, and AFM have been used to evaluate the probes. SEM images show no significant bending of the cantilevers after release, indicating low residual stress, and no stiction between the cantilever and the surface below, which is indicative of the low adhesion energies and hydrophobicity of the UNCD and ta-C surfaces. TEM was used to characterize the apex radius of the tip, and it was found that extremely sharp structures with radii  $< 20$  nm are



feasible. For UNCD, diffraction has shown that the tip material is indeed diamond. Based on the images alone, further optimization is needed. The benefits of oxide sharpening of the silicon molds have been confirmed, without which the tips often form rather large radii above 200 nm. The cantilevers were mounted in an AFM to measure the resonant frequency and quality factor of the probes. From this, the spring constant and Young's modulus have been calculated and compare favorably with the moduli measured previously on these films using other techniques. Furthermore, the UNCD and ta-C AFM probes have been successfully used to image samples in contact mode and high quality topographic and friction images have been repeatedly obtained without noticeable degradation of the image quality. This has led to a successful NSF STTR program in collaboration with Advanced Diamond Technologies, Inc., a start-up company that has invested substantially in commercializing UNCD-based AFM probes. A commercial product is expected to come to market imminently.

Also investigated was the nanotribology and the surface chemistry of hydrogenated diamond-like carbon films, grown by a room temperature deposition technique and then exposed to elevated temperatures ( $\sim 300^\circ\text{C}$ ). The film is grown with the Plasma Immersion Ion Implantation and Deposition (PIID) technique, which is a unique non-line-of-sight method. This film is a hydrogenated DLC with a lower amount of  $\text{sp}^3$ -bonded carbon (30-50%). The nanotribology of carbon-on-carbon interfaces is studied quantitatively with atomic force microscopy (AFM) utilizing silicon AFM tips coated with DLC. The surface chemistry and bonding are probed by total electron yield NEXAFS spectroscopy. The temperature ranges at which structural and chemical changes occur on the surfaces were quantified, and these changes were correlated with specific changes in the nano-scale frictional properties of the interfaces. In particular, F-doped and undoped DLC show moderate changes with annealing, while the Si-doped DLC is impervious to the heating, both in its structure as revealed by NEXAFS, and in the frictional response as probed by AFM. XPS confirmed the corresponding changes (or lack thereof) in the atomic structure of the surface.

Studies of ta-C demonstrate how bonding structure and nanotribological properties are affected by thermal annealing, which is a critical step in the film synthesis required for relieving internal stresses. Specifically, annealing ta-C above  $600^\circ\text{C}$  increases the amount of  $\text{sp}^2$ -bonded carbon near the surface (rehybridization that has a graphitic character), but the work of adhesion between the diamond tip and the ta-C is not affected. Preliminary results indicate that friction forces are modestly reduced by this process. Possible mechanisms that account for this surprising behavior are now being considered.

All of these scientific results are relevant to the AF mission because they provide insights into materials which may be used to alleviate tribological and reliability problems in systems ranging from bearings to MEMS devices. MEMS devices in particular are being actively developed by industry for a range of defense and civilian applications, including precision gyros, miniaturized RF switches and resonators for wireless communication, and biochemical sensors. For bearings and other aerospace components that encounter tribological contact, chameleon coatings developed by AFRL often contain carbon-based constituents. The results presented here help establish the basic science to understand, explain, and predict the behavior of such carbon-based constituents.

## Personnel Supported

Staff Scientist: Dr. Anirudha V. Sumant.

Primary Graduate Students: Dr. David S. Grierson (PhD achieved under this grant), Andrew R. Konicek

Graduates Students Partially Supported: Sean D. O'Connor, Guoqing Ning.

Undergraduate Students: Jason A. Bares, Abraham Spinelli (also supported by a REU fellowship), Graham E. Wabiszewski (also supported by a REU fellowship).

## Publications

**The following peer-reviewed manuscripts have been published thanks to the support of this grant:**

1. Enhanced nucleation, smoothness and conformality of ultrananocrystalline diamond (UNCD) ultrathin films via tungsten interlayers. J.A. Carlisle, N.N. Naguib, J.W. Elam, J. Birrell, J. Wang, D.S. Grierson, B. Kabius, J.M. Hiller, A.V. Sumant, R.W. Carpick, O. Auciello. *Chemical Physics Letters*. v 430, n 4-6, (2006), p 345-50.
2. Surface composition, bonding, and morphology in the nucleation and growth of ultra-thin, high quality nanocrystalline diamond films. R.W. Carpick, A.V. Sumant, P.U.P.A. Gilbert, D.S. Grierson, A.R. Konicek, M. Abrecht, J.E. Butler, T. Feygelson, S.S. Rotter. *Diamond and Related Materials*. v 16, n 4-7, (2007), p 718-724.
3. Small amplitude reciprocating wear performance of diamond-like carbon films: Dependence of film composition and counterface material. J.A. Bares, A.V. Sumant, D.S. Grierson, R.W. Carpick, K. Sridharan. *Tribology Letters*. v 27, n 1, (2007), p 79-88.
4. Tribochemistry and material transfer for the ultrananocrystalline diamond-silicon nitride interface revealed by x-ray photoelectron emission spectromicroscopy. D.S. Grierson, A.V. Sumant, A.R. Konicek, M. Abrecht, J. Birrell, O. Auciello, J.A. Carlisle, T.W. Scharf, M.T. Dugger, P.U.P.A. Gilbert, R.W. Carpick. *Journal of Vacuum Science & Technology B*. v 25, n 5, (2007), p 1700-5.
5. Surface chemistry and bonding configuration of ultrananocrystalline diamond surfaces and their effects on nanotribological properties. A.V. Sumant, D.S. Grierson, J.E. Gerbi, J.A. Carlisle, O. Auciello, R.W. Carpick. *Physical Review B*. v 76, n 23, (2007), p 235429/1-11.
6. Atomic-scale friction on diamond: A comparison of different sliding directions on (001) and (111) surfaces using MD and AFM. G. Gao, R.J. Cannara, R.W. Carpick, and J.A. Harrison, *Langmuir*, v 23, n 10, (2007), p 5394-405.



7. Nanoscale friction varied by isotopic shifting of surface vibrational frequencies. R.J. Cannara, M.J. Brukman, A.V. Sumant, K. Cimat, S. Baldelli, and R.W. Carpick, *Science*, v 318, n 5851, (2007), p 780-3.

8. Synthesis and characterization of smooth ultrananocrystalline diamond films via low pressure bias-enhanced nucleation and growth. Y.C. Chen, X.Y. Zhong, A.R. Konicek, D.S. Grierson, N.H. Tai, I.N. Lin, B. Kabius, J.M. Hiller, A.V. Sumant, R.W. Carpick, O. Auciello. *Applied Physics Letters*. v 92, n 13, (2008), p 133113

9. Origin of ultralow friction and wear in ultrananocrystalline diamond. A.R. Konicek, D.S. Grierson, P.U.P.A. Gilbert, W.G. Sawyer, A.V. Sumant, R.W. Carpick. *Physical Review Letters*. v 100, n 23, (2008), 235502-1-4.

Four more articles are in preparation for submission to peer-reviewed journals.

**The following peer-reviewed, invited review articles have been published thanks to the support of this grant:**

1. Nanotribology of carbon-based materials. D.S. Grierson, R.W. Carpick. *Nano Today*. v 2, n 5, (2007), p 12-21.

2. Recent advances in single-asperity nanotribology. I. Szlufarska, M. Chandross, R.W. Carpick, *J. Phys D: Appl. Phys.* 41 (2008).

### **Interactions/Transitions**

#### ***a. Participation/presentations at meetings, conferences, seminars, etc.***

***Invited talk: Nanotribology: The Science of Friction at the Atomic Scale***

R.W. Carpick

2005 Society of Tribologists and Lubrication Engineers Chicago Section Education Seminar, Mar. 2005.

***Invited talk: Connecting Nanotribology with the Surface Chemistry and Structure of Ultrananocrystalline Diamond and Tetrahedral Amorphous Carbon Thin Films***

R.W. Carpick, A.V. Sumant, D.S. Grierson, G. Ning, J. Birrell, J. Gerbi, J.A. Carlisle, O. Auciello, T.A. Friedmann.

Materials Research Society Spring Meeting, San Francisco, CA, Mar. 2005.

***Invited talk: Multi-length scale experiments and modeling of friction: Connecting micro-device performance with nano-scale contact behavior***

R.W. Carpick, E.E. Flater, C.K. Bora, M.E. Plesha, A.V. Sumant, M.D. Street, M.P. de Boer, Maarten A.D. Corwin, E.D. Reedy

Materials Research Society Spring Meeting, San Francisco, CA, Mar. 2005.



*Invited talk: Nanotribology and Surface Chemistry of Carbon-Based Thin Films Exposed to Elevated Temperatures*

A.V. Sumant, D. S. Grierson, K. Sridharan, T.A. Friedmann, J.P. Sullivan, J. A. Carlisle, O. A. Auciello, and R.W. Carpick

Materials Research Society Spring Meeting, San Francisco, CA, Mar. 2005.

*Poster: Fabrication and Evaluation of Ultrananocrystalline Diamond and Tetrahedral Amorphous Carbon AFM Probes*

Ning Guoqing, A.V. Sumant, D. S. Grierson, J. Birrell, J.E. Gerbi, J. A. Carlisle, O. Auciello, and R. W. Carpick

Materials Research Society Spring Meeting, San Francisco, CA, Mar. 2005.

*Invited talk: Nanotribological Behavior of Carbon-Based Materials*

R.W. Carpick

Society of Tribologists and Lubrication Engineers National Meeting, Las Vegas, NV, May 2005.

*Invited talk: Controlling the Nanotribology of Nanostructured Diamond Films*

R.W. Carpick, D. S. Grierson, A.V. Sumant, E.E. Flater, G. Ning, K. Sridharan, T.A. Friedmann, J.P. Sullivan, J.E. Gerbi, J. Birrell, X. Xiao, O. Auciello, J.A. Carlisle

8th International Conference on Applications of Diamond and Related Materials and the 1st NanoCarbon Joint Conference, Argonne National Laboratory, Argonne, IL, May 2005.

*Invited talk: Morphology and Bonding Structure of Ultra-thin Nanocrystalline Diamond Films*

A.V. Sumant, D.S. Grierson, J.A. Carlisle, J.E. Butler and R.W. Carpick

8th International Conference on Applications of Diamond and Related Materials and the 1st NanoCarbon Joint Conference, Argonne National Laboratory, Argonne, IL, May 2005.

*Talk: Nanotribology of Self-Mated Nanocrystalline and Amorphous Carbon Interfaces*

D. S. Grierson, A.V. Sumant, J.E. Gerbi, J. Birrell, O. Auciello, J.A. Carlisle, K. Sridharan, E.E. Flater, T.A. Friedmann, J.P. Sullivan, R.W. Carpick

8th International Conference on Applications of Diamond and Related Materials and the 1st NanoCarbon Joint Conference, Argonne National Laboratory, Argonne, IL, May 2005.

*Poster: Fabrication and Evaluation of Ultrananocrystalline Diamond and Tetrahedral Amorphous Carbon Atomic Force Microscopy Probes*

Guoqing Ning, A.V. Sumant, D. S. Grierson, and R. W. Carpick, J.E. Gerbi, J. Birrell, X. Xiao, Orlando Auciello, J. A. Carlisle

8th International Conference on Applications of Diamond and Related Materials and the 1st NanoCarbon Joint Conference, Argonne National Laboratory, Argonne, IL, May 2005.

*Invited talk: Towards the Ultimate Tribological Interface: Surface Chemistry and Nanotribology of Nanostructured Carbon Thin Films.*

A.V. Sumant, R.W. Carpick, D.S. Grierson, Guoqing Ning, J.A. Carlisle, O.H. Auciello J., E. Gerbi, J. Birrell, T. A. Friedmann, J. P. Sullivan  
AVS Prairie Chapter Regional Meeting, Northwestern University, Evanston, IL, Jun. 2005.

*Poster: Characterization of Tribo-chemistry in Hard Nanostructured Carbon Materials using X-PEEM Spectromicroscopy.*

A.R. Konicek

SRC User's Meeting, Stoughton, WI, Oct. 2005.

*Invited talk: Friction at the Nanometer Scale: Recent Experimental Advances.*

R.W. Carpick

Kavli Institute for Theoretical Physics, Workshop on "From the Atomic to the Tectonic: From the Atomic to the Tectonic: Friction, Fracture and Earthquake Physics" Santa Barbara, CA, Nov. 2005.

*Talk: Spectromicroscopy studies of tribochemistry: X-PEEM characterization of self-mated wearing in humidity-controlled environments for ultrananocrystalline diamond and tetrahedral amorphous carbon.*

A.R. Konicek

American Chemical Society International Meeting, Atlanta, GA, Mar. 2006.

*Invited talk: Fundamental Studies of Tribochemistry and Wear in Nanostructured Carbon Films Using Nano-scale X-Ray Spectromicroscopy.*

R.W. Carpick

Society of Tribologists and Lubrication Engineers National Meeting, Calgary, AB, Canada, May 2006.

*Invited talk: Combining Nanotribology and X-ray Spectromicroscopy to Understand Wear and Tribochemistry.*

R.W. Carpick

Gordon Research Conference on Tribology, Waterville, ME, Jun. 2006.

*Poster: Spectromicroscopy studies of tribochemistry: X-PEEM characterization of self-mated wear in humidity-controlled environments for tetrahedral amorphous carbon and ultrananocrystalline diamond films.*

A.R. Konicek

Gordon Research Conference on Tribology, Waterville, ME, Jun. 2006.

*Poster: Spectromicroscopy studies of tribochemistry: X-PEEM characterization of self-mated fretting wear in humidity-controlled environments, and nanoscale wear against diamond for tetrahedral amorphous carbon films*

A.R. Konicek

SRMS-5 Conference, Chicago, IL, Jul. 2006.

*Invited talk: Nanotribology of Single Crystal and Nanocrystalline Diamond.*

R.W. Carpick



Diamond-2006 Conference, Estoril, Portugal, Sep. 2006.

***Invited talk: The Role of Atomic Corrugation and Surface Vibrations in the Nanotribology of Carbon-based Systems.***

R.W. Carpick

5th European Science Foundation Nanotribology Workshop, Antalya, Turkey, Sep. 2006.

***Invited talk: The Role of Atomic Corrugation, Crystal Orientation, and Surface Chemical Bonding in the Nanotribology of Carbon-Based Systems.***

R.W. Carpick

American Vacuum Society 53rd International Symposium, San Francisco, CA, Nov. 2006.

***Talk: Spectromicroscopy of tribochemistry: X-PEEM characterization of wear vs. humidity for ultrahard carbon films.***

A.R. Konicek

American Vacuum Society Conference, San Francisco, CA, Nov. 2006.

***Invited talk: Physical Basis of Friction Phenomena for Carbon-Based Systems.***

R.W. Carpick

30th Annual Adhesion Society Meeting, Tampa, FL, Feb. 2007.

***Invited talk: Scanning Probe-Based Nanotribology: Successes, Shortcomings, and Future Directions.***

R.W. Carpick

3rd International Nanotribology Forum Meeting, Hoi An, Vietnam, Mar. 2007.

***Invited talk: Nano-Scale Mechanics and Tribology of Nanocarbon Materials.***

R.W. Carpick

Materials Research Society Spring Meeting, San Francisco, CA, Apr. 2007.

***Talk: Spectromicroscopy of tribochemistry: X-PEEM characterization of wear vs. humidity for ultrahard carbon films.***

A.R. Konicek

Society of Tribologists and Lubrication Engineers Conference, Philadelphia, PA, May 2007.

***Invited talk: Controlling Nanoscale Adhesion and Friction by Directing the Atomic and Molecular Structure of Surfaces.***

R.W. Carpick

Leverhulme, Trust Workshop on Adhesive Interactions between Particles and Surfaces at Micro and Nano-scales, Cardiff University, UK, Jun. 2007.

***Invited talk: Adhesion and Friction at the Nanometer Scale.***

R.W. Carpick

Leverhulme Trust Workshop on Adhesive Interactions Between Particles and Surfaces at Micro and Nano-scales, Cardiff. UK Jul. 2007.

*Invited plenary talk: Nanotribology of Diamond and Amorphous Carbon.*

R.W. Carpick

UK SPM 2007, University of Sheffield, Jul. 2007.

*Invited talk: Nanoscale Friction and Wear of Carbon-Based Materials.*

R.W. Carpick

American Vacuum Society 54th International Symposium, Seattle, WA, Oct. 2007.

*Invited talk: The Study of Nanocarbon Films by and for Atomic Force Microscopy.*

R.W. Carpick

Materials Research Society Fall Meeting, Boston MA, Nov. 2007.

*Invited talk: Adhesion, Friction, and Wear of Diamond.*

R.W. Carpick

GM Research, Dec. 2007.

*Invited talk: Adhesion, Friction, and Wear of Diamond.*

R.W. Carpick

Exxon Mobil, Paulsboro Research Center, Paulsboro, NJ, Jan. 2008.

*Invited talk: Physical Basis of Friction Phenomena for Carbon-Based Systems.*

R.W. Carpick

Adhesion Society Annual Meeting, Tampa FL, Feb. 2008.

*Talk: Evaluating the Environmental Boundaries of Ultrananocrystalline Diamond Thin Film Coatings.*

M.A. Hamilton

Materials Research Society Conference, San Francisco, CA, Mar. 2008.

*Talk: Environmental dependence of tribochemical wear for ultrahard carbon films.*

A.R. Konicek

Society for Tribologists and Lubrication Engineers Conference, Cleveland, OH, May 2008.

*Talk: Tribological Response of Ultrananocrystalline Diamond Coatings to Varying Environments.*

M.A. Hamilton

Society for Tribologists and Lubrication Engineers Conference, Cleveland, OH, May 2008.

*Poster: Ultralow friction, tribochemical wear, and the humidity-induced frictional transition of ultrananocrystalline diamond interfaces.*

A.R. Konicek

Center for Nanoscale Materials User's Meeting, Argonne, IL, May 2008.

*Poster: Ultra low friction, tribochemical wear, and the humidity-induced frictional transition of ultrananocrystalline diamond interfaces.*

A.R. Konicek



Gordon Research Conference on Tribology, Waterville, ME, Jul. 2008.

*Upcoming: Nanotribology of Carbon: How the Topmost Atoms Matter.*

R.W. Carpick

2008 International Conference on Nanoscience + Technology, Keystone CO, Jul. 2008.

*Upcoming: Origin Of Ultralow Friction and Wear In Ultrananocrystalline Diamond.*

A.R. Konicek

2008 Diamond Conference, Sitges, Spain, Sep. 2008.

*Upcoming: Connections Between the Surface Science and Tribology of Carbon-Based Material.*

R.W. Carpick

Eastern Analytical Society, Somerset NJ, Nov. 2008.

***b. Consultative and advisory functions to other laboratories and agencies, especially Air Force and other DoD laboratories.***

This work included substantial, extended interactions with Argonne National Laboratories (who have been supplying UNCD and DLC samples which were characterized in detail) and Sandia National Laboratories (supplying ta-C and polysilicon MEMS devices which were characterized extensively).

This work also instigated interactions with AFRL (Dr. Andrey Voevodin, Dr. Chris Muratore) to investigate nano-scale features on worn "chameleon" coatings. Dr. Voevodin was invited to give a talk at UW-Madison in the spring, 2006 and Dr. Chris Muratore was invited to give a presentation at a Materials Research Society Symposium entitled "Nanotribology: Impact for Materials and Devices" organized by the PI (Carpick) and supported by AFOSR funds.

There have been informal interactions with the tribology group Aerospace Corporation to discuss common interests, particularly in carbon-based thin film tribological and surface science studies.

***c. Transitions.***

The discoveries made on carbon-based films have potentially wide-spread applications in MEMS. The results have been discussed with representatives from Boeing, Honeywell, Freescale Semiconductor, MEMtronics, Innovative Micro Technologies, and Peregrine Semiconductor. There is clear potential to apply the results to the design of MEMS components, specifically high-speed, small gyros for precision navigation, and high speed ohmic and capacitive RF MEMS switches. MEMtronics is currently investigating the integration of UNCD into its RF switch design and has demonstrated impressive functionality. This led to DARPA funding within the Microsystems Technology Office.

As well, there have been significant interactions with Advanced Diamond Technologies Inc. (Urbana, IL). The technical contact is Dr. John Carlisle, CTO. ADT is pursuing the development of AFM cantilevers fabricated entirely from UNCD, which was accomplished in this work. This

success, and the demonstration of the excellent tribological properties of UNCD, have been the key drivers. A STTR proposal, led by ADT with members of this group as collaborators was submitted to NSF, successfully funded for Phase I and then Phase II support. The commercial production of UNCD-based cantilevers is a likely successful outcome of work supported by this grant.

There have also been interactions with IBM Research (Zürich). The technical contacts are Urs Dürig, Michel Despont, Mark Lantz, and Bernd Gotsmann. They work on the "Millipede" project, which is a novel information storage concept based on atomic force microscope cantilevers. IBM microfabricates unique cantilevers with built-in heaters which are capable of thermomechanical data storage. The technique has the potential to increase information storage density compared with the best hard drives by a factor of at least 100. Tribological issues, namely wear of the tip, limit the performance and lifetime of the device. Collaborative research is ongoing to apply carbon-based coatings to the tips to increase their robustness. The initial tests with these coatings are encouraging and the work is continuing.

#### **New discoveries, inventions, or patent disclosures**

Discoveries are discussed above. Two patent disclosures have been submitted to UW's patent agent and accepted by them for submission to the U.S. Patent Office. One Patent Application has now been submitted, and the other is in preparation.

AFM microcantilevers made entire of, or coated with, ultrahigh performance diamond-based materials were fabricated. The specific materials used were ta-C, UNCD, and DLC. AFM is a widely used tool in research and industry, and minimizing the wear of the nano-scale tips used in AFM is a critical challenge. UNCD, DLC, and ta-C have excellent mechanical properties that are ideally suited for AFM, namely low wear, low friction, high hardness, and chemical inertness. The first successful batch fabrication of monolithic probes made from ta-C and UNCD was achieved, and silicon AFM cantilevers have been successfully coated with DLC and ta-C. This work showed that reliable measurements can be conducted with them. This technology has the potential to revolutionize the use of AFM for imaging, metrology, nanomachining, nanomanufacturing, nanomechanical testing, and nanomechanical data storage. This has sparked collaborations with industry (ADT, and IBM).

#### **Honors/Awards**

R.W. Carpick, "Non-Tenured Faculty Award", 3M Corporation, March 2005.

R.W. Carpick, awarded tenure & promoted to Associate Professor, University of Wisconsin-Madison, June 2005.

R.W. Carpick, Invited Member, Proposal Review Committee of the Molecular Foundry, Lawrence Berkeley National Laboratory (since 2005).



R.W. Carpick, Invited Participant, Kavli Institute of Theoretical Physics, University of California, Santa Barbara: "From the Atomic to the Tectonic: Friction, Fracture and Earthquake Physics" (to be held Nov.-Dec. 2005).

R.W. Carpick, named University of Pennsylvania Director of the Nanotechnology Institute (<http://www.nanotechinstitute.org>)

A.V. Sumant, elected Program Chair, MEMS and NEMS Technical Group, American Vacuum Society, 2005.

A.R. Konicek, Physical Review Letters article selected "Editor's Choice" in Science, 2008.

A.R. Konicek, Best Poster, 2008 Gordon Research Conference on Tribology.